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最終頁に続く

(54) 【発明の名称】 多孔性炭素質成形板用プリプレグシート

(57) 【要約】

【目的】 表面平滑性に優れた多孔性炭素質成形板を得る。

【構成】 炭素繊維と固形フェノール樹脂を主成分に、エポキシ樹脂を副成分としてそれを水性媒体中に分散したスラリーを抄紙したプリプレグシート。当該シートは圧縮成形されたのち、焼成して多孔性炭素質成形板とされる。

【効果】 有機繊維をさらに併用して得たプリプレグシートから得られた炭素質成形板は、表面平滑性のみならず燃料電池電極板に要求される多孔性も有していた。

## 【特許請求の範囲】

【請求項1】 炭素繊維（A）と固形フェノール樹脂（B）とを主成分とし抄紙して得られるシートであって、圧縮成形、焼成に供して多孔性炭素質成形板とする多孔性炭素質成形板用アリアレグシートにおいて、樹脂（B）に副成分としてエポキシ樹脂（C）を併用したことを特徴とする多孔性炭素質成形板用アリアレグシート。

【請求項2】 エポキシ樹脂（C）にさらに有機繊維（D）を併用する請求項1記載のアリアレグシート。

【請求項3】 炭素繊維（A）が、ピッチ系炭素繊維である請求項1記載のアリアレグシート。

【請求項4】 固形フェノール樹脂（B）が、重量平均分子量500～10万の範囲のものである請求項1記載のアリアレグシート。

【請求項5】 有機繊維（D）が、フィブリル化していない有機繊維である請求項2記載のアリアレグシート。

【請求項6】 固形フェノール樹脂（B）重量に対するエポキシ樹脂（C）重量が、5～50重量%の範囲である請求項1記載のアリアレグシート。

【請求項7】 炭素繊維（A）に対する有機繊維（D）の割合が、5重量%から50重量%の範囲である請求項2記載のアリアレグシート。

【請求項8】 炭素繊維（A）と固形フェノール樹脂（B）とを主成分とし抄紙して得られるシートであって、圧縮成形、焼成に供して多孔性炭素質成形板とする多孔性炭素質成形板用アリアレグシートにおいて、樹脂（B）に副成分としてエポキシ樹脂（C）を併用したことを特徴とする磷酸型燃料電池電極板用アリアレグシート。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は、圧縮成形、焼成して用いる多孔性炭素質成形板を得るのに好適な炭素繊維と固形フェノール樹脂を主成分として抄紙したアリアレグシートに関する。

## 【0002】

【従来の技術】多孔性炭素質成形板は種々の方法で製造されている。例えば、炭素繊維のペーパー、フェルト、クロス等の炭素繊維集合体に熱硬化性樹脂を含浸、乾燥させ樹脂を半硬化状態（Bステージ化）のアリアレグと成し、該アリアレグを圧縮成形等によって成形し、不活性雰囲気下で焼成することによって得られる。このように最終工程で焼成するため、炭化によって残る収率（残炭率）が高いのが望ましく、一般的にはフェノール樹脂を用いたアリアレグシートが用いられる。

【0003】特に均質な薄板を製造する際、炭素繊維を連続した長繊維の形態で用いると、繊維を均質に分散させるのが難しく、繊維密度の不連続な部位が発生し方向性の無い均一な薄板を成形するのが非常に困難であり、

通常は炭素繊維の短繊維集合体、即ちペーパー、不織布、或いはニードルパンチしたフェルト等が用いられる。

【0004】ところが、均質な薄板を製造する際に用いられる炭素繊維の短繊維集合体、即ちペーパー、不織布、或いはニードルパンチしたフェルト等は、その目的から単位面積当たりの重量が小さく、且つその力学強度も弱いので、アリアレグ化工程における樹脂比率やBステージ化の制御が難しく、品質や歩留まりのトラブルが生じ易い。

【0005】この問題を解決するひとつの手段としてフェノール樹脂をバインダーとする湿式抄紙法による炭素繊維ペーパーが考えられる。この方法によれば炭素繊維ペーパー中にあらかじめ所定量のフェノール樹脂をバインダーとして含ませることが可能であり、アリアレグ工程が不要となる。

【0006】しかしながらフェノール樹脂はほとんどの場合、フリーフェノールが含まれ湿式抄紙における排水処理に問題があり、現実的には実機レベルでの抄紙はほとんど不可能にちかい。

【0007】これに対し、固形フェノール樹脂は、フリーフェノールを含まないものが可能で、抄紙工程での排水処理の問題もなく実機レベルでの抄紙が可能である。

## 【0008】

【発明が解決しようとする課題】しかしながら、固形フェノール樹脂は、熱溶解時の粘度が高いために、熱成形工程に於いては均質な成形板を作るのが非常に困難で、樹脂の分散性に劣り、表面平滑性の悪い板ができやすいという欠点があった。

## 【0009】

【課題を解決する為の手段】本発明者らは、これらの課題を解決すべく鋭意研究した結果、炭素繊維と固形フェノール樹脂の主成分以外に第3成分を添加して抄紙したアリアレグシートが、多孔性炭素質成形体の製造における上記欠点を解決できる事を見だし、本発明を完成するに至った。

【0010】即ち本発明は、炭素繊維（A）と固形フェノール樹脂（B）とを主成分とし抄紙して得られるシートであって、圧縮成形、焼成に供して多孔性炭素質成形板とする多孔性炭素質成形板用アリアレグシートにおいて、樹脂（B）に副成分としてエポキシ樹脂（C）を併用したことを特徴とする多孔性炭素質成形板用アリアレグシート及びそのアリアレグシートを圧縮成形、焼成した多孔性炭素質成形板を提供するものである。

【0011】本発明で用いられる炭素繊維（A）は、特に限定されるものではないが、例えばPAN系炭素繊維、ピッチ系炭素繊維、あるいは各種有機繊維を出発原料とする炭素繊維、またはこれらを黒鉛化した黒鉛繊維のいずれも使用出来る。これらの繊維（A）は、目的に応じて単独、あるいは組み合わせて使われる。

【0012】繊維(A)の繊維径は、特に限定されないが、その強度および取扱性から、5~25 $\mu$ mの範囲にあるものが望ましい。アスペクト比(繊維長/繊維径)は、例えば抄紙されるシートの強度およびその抄紙工程における分散性を考慮すると50以上、好ましくは50~10000の範囲が良い。

【0013】本発明で用いられる固形フェノール樹脂(B)としては、常温で固形である公知慣用のものがいずれも使用出来、特に限定されるものではない。熱溶融又は熱硬化タイプのものとしては、例えばレゾール樹脂、ノボラック樹脂等がある。前者は、一般的に樹脂単独で熱硬化する自硬化型であり、後者は、それ自体では硬化性を有さないで、一般的に硬化剤が併用される。樹脂(B)としては、重量平均分子量500~10万のものが残炭率の観点からすると好ましい。

【0014】勿論、自硬化型のもの場合でも、必要に応じて硬化剤、あるいは硬化助剤を添加して硬化条件を緩和することはよく行われる。硬化剤としては、例えばヘキサメチレンテトラミンやポリアミド樹脂等が使用できる。

【0015】本発明で用いられるエポキシ樹脂(C)は、例えば前記樹脂(B)に可塑性を付与するために添加される。本発明において、前記基本成分に添加することが出来るエポキシ樹脂としては、例えばビスフェノールA系エポキシ樹脂、ノボラック型エポキシ樹脂やその他のグリシジルエーテル系エポキシ樹脂及びその変成物が挙げられる。中でも樹脂(C)としては、融点60℃以上200℃以下好ましくは80℃以上150℃以下のものが好ましい。

【0016】樹脂(C)の使用量は、特に限定されないが、例えば樹脂(B)使用量に対して5重量%から50重量%となる様に用いることが望ましい。この範囲における添加に対しては、可塑性効果もレベルアップすることなく、後述する焼成されて得られる成形板の強度も良好である。

【0017】固形フェノール樹脂(B)とエポキシ樹脂(C)との合計使用量は、炭素繊維(A)と固形フェノール樹脂(B)との合計に対して、通常5重量%~90重量%の範囲であり、好ましくは30重量%~70重量%の範囲が良い。これらの範囲内であれば、アブリグシート自体の強度も充分であり、成形するのも容易である。またこの範囲であれば、このアブリグシートから最終的に得られる多孔性炭素質成形板としての力学強度も良好となる。

【0018】本発明の多孔性炭素質成形板用アブリグシートを得るに当たっては、焼成後の炭素質成形板のガス透過度を充分な値となる様にするために、さらに有機繊維(D)をも併用することが出来る。

【0019】繊維(D)としては、公知慣用のものがいずれも使用出来、特に限定されるものではないが、例え

ばレーヨン、ポリエステル繊維、ポリプロピレン繊維、ポリエチレン繊維、アラミド繊維などが使用出来る。繊維(D)としては、フィブリル化していない非開繊の有機繊維が好ましい。フィブリル化している有機繊維、例えば木材パルプ繊維は、それを用いて最終的に得られたアブリグシートを成形焼成すると、炭化後に皮膜構造を形成し、ガス透気度が低下する傾向がある。

【0020】繊維(D)は、炭素繊維(A)と固形フェノール樹脂(B)合計重量の100重量部に対して、通常10~60重量部、中でも後工程での成形圧の観点から10~40重量部とするのが好ましい。

【0021】これら繊維(A)、(D)には、必要に応じて熱特性、力学特性をより向上させるために、例えばセラミック繊維、炭化珪素繊維、窒化珪素繊維などの各種無機繊維や、黒鉛粉末などのフィラーも添加する事も可能である。

【0022】本発明のアブリグシートは、上記繊維(A)、樹脂(B)、樹脂(C)及び必要に応じて繊維(D)を用いて抄紙することにより容易に製造することが出来る。抄紙方法としては、乾式法及び湿式法のいずれも採用するが、湿式法が均一性の点で好ましい。

【0023】この湿式抄紙法としては、例えば繊維(A)、樹脂(B)、樹脂(C)及び必要に応じて繊維(D)、各種フィラーを、必要に応じて分散助剤が加えられた水性媒体中に加え、均一となる様に分散し、スラリーにしてから、傾斜型網で漉いて、水性媒体のみを除去して湿式不織紙ウェブを得、それを乾燥することにより、アブリグシートとする方法が採用出来る。

【0024】また、例えば繊維(A)及び必要に応じて繊維(D)を、必要に応じて分散助剤が加えられた水性媒体中に加え、均一となる様に分散し、スラリーにしてから、傾斜型網で漉いて、水性媒体のみを除去して湿式不織紙ウェブを得、それに樹脂(B)と樹脂(C)とを必須成分として含む組成物を熱時含浸してから乾燥する方法も採用出来る。

【0025】分散助剤としては、公知慣用の保護コロイド、界面活性剤等が使用できるが、例えばポリエチレンオキサイド、ポリオキシアルキレンモノフェノールエーテル、アルキルベンゼンスルホン酸金属塩、ポリビニルアルコール、自己乳化性アクリル樹脂や自己乳化性エポキシ樹脂等を使用することが出来る。

【0026】また、樹脂粉末には、抄紙時の歩留まりを向上させるために各種高分子定着剤を使用することが望ましい。これら定着剤としては、例えばポリアクリルアミド樹脂、硫酸アルミニウム、ポリ塩化アルミニウム等が挙げられる。

【0027】乾燥したアブリグシートを得るに当たっては、例えば熱風乾燥、遠赤外線照射、マイクロ波照射等の方法が採用できる。シート自体の粘着性だけでなく、高度に乾燥を行うと、

後述の圧縮成形に所望の形状に賦型するのが難しくなる。緻密な炭素質成形板を得るに当たっては、この乾燥条件を吟味して行う。

【0028】本発明のアリアレグシートは、如何なる厚みのものでも良いが、例えば0.2~1.2mmのものである。1m<sup>2</sup>当たりの重量は、通常75~300gである。

【0029】この様にして得られたシートは、圧縮成形して賦型してから焼成することにより、例えば多孔性炭素質成形板とすることが出来る。この圧縮成形では、公知慣用の金型を用いて、必要に応じて熱も同時に加えて成形が行われる。勿論、金型内を真空中に脱気して成形する真空圧縮成形法も採用出来る。

【0030】いずれの方法に於いてもシートを一枚あるいは複数枚積層して成形するが、この際、目的に応じて異種のシートをそれと積層して成形することも可能である。

【0031】次いで、前記成形で得られた成形板は、焼成することにより、炭素質でかつ多孔性の成形板とすることが出来る。焼成の条件は、特に制限されるものではないが、例えば窒素、アルゴン等の不活性ガス雰囲気中、又は真空中で1000~3000℃の温度で焼成する事により炭素質の成形板を得ることができる。目的によっては更に2000~3000℃の温度で焼成する事により黒鉛化することも可能である。

【0032】このようにして得られた多孔性炭素質成形板は、公知慣用の種々の用途に採用出来る。具体的には、例えば各種電極材料、セパレーター、断熱材等の用途が挙げられる。

【0033】フィブリル化していない有機繊維を用い、固形フェノール樹脂重量に対するエポキシ樹脂重量が、5~50重量%の範囲であり、かつ炭素繊維に対する前記有機繊維の割合が、5重量%から50重量%の範囲である本発明のシートを用いて得た炭素質成形板は、その特性が電極板に要求される、表面平滑性、強度、ガス透気度等を満足し、電極板用として非常に有用な素材となる。特に磷酸型燃料電池電極板としての仕様値と良く一致する。

【0034】これら上記した要求性能は、電極板固有の

もので、単に成形して得られた成形板からは直接予想することが出来ないものであることは、多言を要さないことである。

【0035】

【実施例】次に本発明を実施例により詳細に説明する。以下、特に断りのない限り、「部」及び「%」は、いずれも重量基準とする。

【0036】[実施例1]アスペクト比460のピッチ系炭素繊維[(株)ドナック製ドナカーボ、繊維径13.5μm]と、ポリブチレンテレフタレート(PBT)繊維[1d×3mm]とを比率4/6(重量比)で、合計繊維濃度0.2%にて水に分散し、そこに自硬化型フェノール樹脂(重量平均分子量12000)と、ビスフェノールAジグリシジルエーテル型エポキシ樹脂(融点100℃、エポキシ当量950)を添加してスラリーを調製した。

【0037】各成分の含有量をフェノール樹脂粉末20部、エポキシ樹脂粉末1部、炭素繊維200部、PBT繊維10部としてシートの抄造を行った。この時のフェノール樹脂に対するエポキシ樹脂添加量は5重量%、炭素繊維に対する有機繊維含有量は5重量%であった。

【0038】樹脂粉末の歩留まりを向上するために、上記スラリーに、カチオン性ポリアクリルアミドを対繊維合計0.3%添加して、樹脂成分の繊維基材への定着向上を図った。このスラリーを傾斜型金網不織布製造装置にて、抄紙して、湿潤した湿式ウェブを得た。

【0039】このウェブを、140℃の熱風で4分間乾燥させ、非粘着性の175g/m<sup>2</sup>のアリアレグシートを得た。

【0040】本シートを2枚重ねて圧縮成形(170℃、圧力7kg/cm<sup>2</sup>、20分間)し、更に真空中で2000℃で焼成し、黒鉛板を得た。得られた黒鉛板の物性値を表1に示した。

【0041】[比較例1]エポキシ樹脂粉末1部を用いない以外は、実施例1と同様な条件で抄紙を行ってアリアレグシートを得、次いで同様な条件で焼成を行って黒鉛板を得た。得られた黒鉛板の物性値を表1に示した。

【0042】

【表1】

表1

	実施例1	比較例1
坪量 (g/m <sup>2</sup> )	170	168
密度 (g/cm <sup>3</sup> )	0.28	0.27
厚み (mm)	0.42	0.42
かさ密度 (g/cm <sup>3</sup> )	0.42	0.42
曲げ強度 (kgf/mm <sup>2</sup> )	1.5	1.4
黒鉛板の表面状態 (目視)	○	×
磷酸型燃料電池電極板としてのガス透気度	○	○

【0043】これらの評価項目の実際の測定条件は次の通りである。

坪量：JIS P8124に準拠して測定した。

密度：JIS P8118に準拠して測定した。

厚み：マイクロメーターを使用して測定。

かさ密度：JIS R7212に準拠して測定した。

曲げ強度：同上。

表面状態（表面平滑性）：目視にて判定した。

ガス透気（過）度：改良ガーレー法により測定した。

【0044】実施例1と比較例1との対比からわかる通り、実施例1のアプレグは、エポキシ樹脂を併用している30ので、その可塑性効果により、熱成形時において樹脂成分の分散性・流動性が良好であり、均質な成形板が得られている。従って、それを焼成した黒鉛板も表面\*

20\*平滑性に優れたものとなっていることがわかる。

【0045】[実施例2～3]表2に記載の通りのスラリーを各々上記したのに従って調製し、それを用いる以外は、実施例1と同様な条件で抄紙を行ってアプレグシートを得、次いで同様な条件で焼成を行って黒鉛板を得た。得られた黒鉛板の物性値を表2に示した。

【0046】尚、フェノール樹脂、エポキシ樹脂及び炭素繊維は実施例1で用いたのと同じのものを、一方、レーヨン繊維としては、大和紡績（株）製ダイワボウレーヨン（1d×3mm）を用い、木材パルプ繊維としては、晒しパルプ（広葉樹クラフトパルプ、フリーネス600cc）を用いた。

【0047】

【表2】

表2

		単位	実施例2	実施例3
樹脂粉末	(B) フェノール樹脂	部	85	
	(C) エポキシ樹脂	部	20	
	(C)/(B)×100	%	24	
繊維基材	(A) 炭素繊維	部	60	
	(D) 有機繊維 種類 含有量	部	レーヨン繊維 10	木材パルプ 10
	(D)/(A)×100	%	17	
シート物性	坪量 密度	g/m <sup>2</sup> g/cm <sup>2</sup>	175 0.34	176 0.34
黒鉛板の物性	厚み	mm	0.40	0.40
	かさ密度	g/cm <sup>3</sup>	0.51	0.51
	曲げ強度	kgf/mm <sup>2</sup>	2.0	2.0
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	×

【0048】実施例3の黒鉛板は、ガス透気性を有してしたが、磷酸型燃料電池電極板用としては、不十分なものであった。

【0049】[実施例4～5] 表3に記載の通りのスラリーを各々上記したのに従って調製し、それを用いる以外は、実施例1と同様な条件で抄紙を行ってアリアレグシートを得、次いで同様な条件で焼成を行って黒鉛板を得た。得られた黒鉛板の物性値を表3に示した。 \*

\* 【0050】尚、フェノール樹脂、エポキシ樹脂及び炭素繊維は実施例1で用いたのと同じのものを、レーヨン繊維は実施例2で用いたのと同じのものを、一方、ポリプロピレン (PP) 繊維としては、1d×3mmのもを用いた。

【0051】

【表3】

表3

		単位	実施例4	実施例5
樹脂粉末	(B) フェノール樹脂	部	85	100
	(C) エポキシ樹脂	部	42	20
	(C)/(B)×100	%	49	20
繊維基材	(A) 炭素繊維	部	60	15
	(D) 有機繊維 種類 含有量	部	PP繊維 30	レヨン繊維 3
	(D)/(A)×100	%	50	20
シート物性	坪量 密度	g/m <sup>2</sup> g/cm <sup>2</sup>	160 0.35	174 0.40
黒鉛板の物性	厚み	mm	0.39	0.40
	かさ密度	g/cm <sup>3</sup>	0.52	0.60
	曲げ強度	kgf/mm <sup>2</sup>	1.9	1.3
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	○

【0052】

\*フェノール樹脂にエポキシ樹脂を併用しているので、最

【発明の効果】本発明では、多孔性炭素質成形板用プリ  
ブレグシートを得るに当たって、従来の樹脂成分である\*30  
最終的に優れた表面平滑性の多孔性炭素質成形板が得られ  
るという格別顕著な効果を奏する。

フロントページの続き

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(21)Application number : 06-294582 (71)Applicant : HONSHU PAPER CO LTD  
DAINIPPON INK & CHEM INC  
(22)Date of filing : 29.11.1994 (72)Inventor : YOKOYAMA HIDEKUNI  
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## (54) PREPREG SHEET FOR PLATY POROUS CARBONACEOUS MOLDING

### (57)Abstract:

PURPOSE: To obtain a platy porous carbonaceous molding excellent in surface smoothness.

CONSTITUTION: A prepreg sheet is obtd. by dispersing carbon fibers and a solid phenol resin, as the main components, and an epoxy resin, as a subsidiary component, in an aq. medium and forming the resulting slurry into a sheet. The prepreg sheet is compression molded and baked to give a platy porous carbonaceous molding. A prepreg sheet obtd. by using an org. fiber material in addition to the above-mentioned components gives a platy carbonaceous molding having not only surface smoothness but also porosity required of an electrode plate of a fuel cell.

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3. In the drawings, any words are not translated.

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CLAIMS

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[Claim(s)]

[Claim 1] The prepreg sheet for porous carbonaceous fabrication boards characterized by using an epoxy resin (C) together as an accessory constituent to a resin (B) in the prepreg sheet for porous carbonaceous fabrication boards which is a sheet which considers as a principal component and is obtained by carrying out paper making, presents compression molding and baking with a carbon fiber (A) and solid phenol resin (B), and uses them as a porous carbonaceous fabrication board.

[Claim 2] The prepreg sheet according to claim 1 which uses organic fiber (D) together further to an epoxy resin (C).

[Claim 3] The prepreg sheet according to claim 1 whose carbon fiber (A) is a pitch based carbon fiber.

[Claim 4] The prepreg sheet according to claim 1 whose solid phenol resin (B) is the thing of the range of weight average molecular weight 500-100,000.

[Claim 5] The prepreg sheet according to claim 2 whose organic fiber (D) is organic fiber which is not fibrillating.

[Claim 6] The prepreg sheet according to claim 1 whose epoxy resin (C) weight to a solid phenol resin (B) weight is 5 - 50% of the weight of a range.

[Claim 7] The prepreg sheet of the organic fiber (D) to a carbon fiber (A) according to claim 2 which is 5 to 50% of the weight of a range comparatively.

[Claim 8] The prepreg sheet for phosphoric acid type fuel cell electrode boards characterized by using an epoxy resin (C) together as an accessory constituent to a resin (B) in the prepreg sheet for porous carbonaceous fabrication boards which is a sheet which considers as a principal component and is obtained by carrying out paper making, presents compression molding and baking with a carbon fiber (A) and solid phenol resin (B), and uses them as a porous carbonaceous fabrication board.

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[Translation done.]

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the prepreg sheet which made the principal component a suitable carbon fiber to obtain the porous carbonaceous fabrication board calcinated [ is pressed and ] and used and solid phenol resin, and carried out paper making.

[0002]

[Description of the Prior Art] The porous carbonaceous fabrication board is manufactured by various methods. For example, sink into the carbon fiber aggregates, such as a paper of a carbon fiber, felt, and a cross, they are made to dry thermosetting resin, a resin is accomplished with the prepreg of a semi-hardening state (formation of B stage), this prepreg is fabricated with compression molding etc., and it is obtained by calcinating under an inert atmosphere. Thus, in order to calcinate by the final process, desirably [ a thing with the high yield (actual carbon ratio) which remains by carbonization ], and generally the prepreg sheet using phenol resin is used.

[0003] If a carbon fiber is used with the form of the continuous continuous glass fiber in case homogeneous sheet metal is manufactured especially, it will be difficult to distribute fiber homogeneously, it will be very difficult to fabricate the uniform sheet metal which a part with discontinuous fiber density occurs and does not have directivity, and the staple-fiber aggregate of a carbon fiber, i.e., a paper, a nonwoven fabric, or the felt that carried out needle punch will usually be used.

[0004] However, since the weight per unit area is small and the dynamics intensity also has it from the purpose, control of the thing resin ratio in a prepreg-ized process or the formation of B stage is difficult for the staple-fiber aggregate of the carbon fiber used in case homogeneous sheet metal is manufactured, i.e., a paper, a nonwoven fabric, or the felt that carried out needle punch, and the trouble of quality or the yield tends to produce it. [ weak ]

[0005] The carbon fiber paper by the wet paper-making method which uses phenol resin as a binder as one means to solve this problem can be considered. According to this method, it is possible to include the phenol resin of the specified quantity as a binder beforehand in a carbon fiber paper, and a prepreg process becomes unnecessary.

[0006] However, in almost all cases, a free phenol is contained, phenol resin has a problem in the waste water treatment in wet paper making, and paper making in system level is almost actually next to impossible.

[0007] On the other hand, what does not contain a free phenol is possible for solid phenol resin, it does not have the problem of the waste water treatment in a paper-making process, and paper making in system level is possible for it.

[0008]

[Problem(s) to be Solved by the Invention] However, since solid phenol resin had the high viscosity at the time of a thermofusion, it was very difficult to make a homogeneous forming board in a thermoforming process, was inferior to the dispersibility of a resin in it, and had the fault that it could be tended to do the bad board of surface smooth nature.

[0009]

[Means for Solving the Problem] As a result of inquiring wholeheartedly that these technical problems should be solved, this invention persons find out that the prepreg sheet which added and carried out paper making of the 3rd component in addition to the principal component of a carbon fiber and solid phenol resin can solve the above-mentioned fault in manufacture of a porous carbonaceous Plastic solid, and came to complete this invention.

[0010] Namely, this invention is a sheet which makes a principal component a carbon fiber (A) and solid phenol resin (B), and is obtained by carrying out paper making, and is set on the prepreg sheet for porous carbonaceous fabrication boards which presents compression molding and baking with and which is used as a porous carbonaceous fabrication board. The porous carbonaceous fabrication board which pressed the prepreg sheet for porous carbonaceous fabrication boards characterized by using an epoxy resin (C) together as an accessory constituent to a resin (B) and ] the prepreg sheet, and was calcinated is offered.

[0011] Although especially the carbon fiber (A) used by this invention is not limited, either a PAN system carbon fiber, a pitch based carbon fiber the carbon fiber that uses various organic fiber as a start raw material or the graphite fiber which graphitized these can be used for it, for example. these fiber (A) -- the purpose -- responding -- independence -- or it is used, combining

[0012] Although especially the diameter of fiber of fiber (A) is not limited, what is in the range of 5-25 micrometers from the intensity and handling nature is desirable. an aspect ratio (fiber length/diameter of fiber) -- for example, when the dispersibility in the intensity and its paper-making process of the sheet by which paper making is carried out is taken into

consideration, the range of 50-10000 is [ 50 or more ] preferably good

[0013] As solid phenol resin (B) used by this invention, each thing of the well-known common use which is a solid can use it in ordinary temperature, especially it is not limited. As a thermofusion or heat-curing type thing, there are a resol resin, a novolak resin, etc., for example. The former is the self-hardening type which can generally heat-harden by the resin independent, and since the latter does not have hardenability in itself, generally a curing agent is used together. As a resin (B), the thing of weight average molecular weight 500-100,000 is desirable, considering the viewpoint of an actual carbon ratio.

[0014] Of course, adding a curing agent or a hardening assistant if needed, and easing hardening conditions also by the case of a self-hardening type thing, is often performed. As a curing agent, a hexamethylenetetramine, polyamide resin, etc. can be used, for example.

[0015] The epoxy resin (C) used by this invention is added in order to give plasticity to for example, the aforementioned resin (B). In this invention, the bisphenol A system epoxy resin, a novolak type epoxy resin, other glycidyl ether system epoxy resins, and the conversion object of those are mentioned as an epoxy resin which can be added to the aforementioned fundamental component, for example. As a resin (C), 80-degree-C or more thing 150 degrees C or less is preferably [ 200 degrees C or less of 60 degrees-C or more melting points ] desirable especially.

[0016] Although especially the amount of the resin (C) used is not limited, it is desirable to use so that it may become 50 % of the weight from 5 % of the weight, for example to the amount of the resin (B) used. The intensity of the forming board which is mentioned later and which is calcinated and is obtained is also good, without carrying out the level off also of the plasticization effect to the addition in this range.

[0017] The amount of the sum total used of solid phenol resin (B) and an epoxy resin (C) is usually 5 % of the weight - 90% of the weight of a range to the sum total of a carbon fiber (A) and solid phenol resin (B), and 30 % of the weight - 70% of the weight of its range is preferably good. If it is within the limits of these, the intensity of the prepreg sheet itself is also enough and fabricating is also easy. Moreover, if it is this range, the dynamics intensity as a porous carbonaceous fabrication board finally obtained from this prepreg sheet will also become good.

[0018] In order to make the gas transmittance of the carbonaceous fabrication board after baking become sufficient value in obtaining the prepreg sheet for porous carbonaceous fabrication boards of this invention, organic fiber (D) can be further used together.

[0019] Although each thing of well-known common use can use it, especially it is not limited as fiber (D), rayon, a polyester fiber, a polypropylene fiber, a polyethylene fiber, an aramid fiber, etc. can be used, for example. As fiber (D), the organic fiber of the non-filamentation which is not fibrillating is desirable. When the organic fiber which is fibrillating, for example, wood pulp fiber, carries out forming baking of the prepreg sheet finally obtained using it, it forms coat structure after carbonization and has the inclination for gas air permeability to fall.

[0020] As for fiber (D), it is desirable to usually consider as 10 - 40 weight section from a viewpoint of the moulding pressure in a back process also in 10 - 60 weight section to the 100 weight sections of a carbon fiber (A) and a solid phenol resin (B) sum total weight.

[0021] It is also possible to also add various inorganic fibers, such as a ceramic fiber, a silicon carbide fiber, and silicon-nitride fiber, and fillers, such as a graphite powder, to them, in order to raise a heat characteristic and a dynamics property more to these fiber (A) and (D) if needed.

[0022] The prepreg sheet of this invention can be easily manufactured by carrying out paper making using fiber (D) the above-mentioned fiber (A), a resin (B), a resin (C), and if needed. As the paper-making method, both dry process and a wet method are adopted, and \*\* of a wet method is desirable in respect of homogeneity.

[0023] As this wet paper-making method, fiber (A), a resin (B), a resin (C), and the need are accepted, for example. Fiber (D), After it distributes [ be / under / water medium / by which it was added to the distributed assistant if needed ] adding / it ] so that it may become uniform, and making various fillers into a slurry, the method of using as a prepreg sheet is employable by an inclined type network's removing only \*\*\*\*\* and a water medium, obtaining a wet nonwoven web, and drying it.

[0024] Moreover, it adds into the water medium by which fiber (D) was added to the distributed assistant if needed fiber (A) and if needed, for example. After it distributes so that it may become uniform, and making it a slurry, an inclined type network removes only \*\*\*\*\* and a water medium, a wet nonwoven web is obtained, and the method of drying the constituent which contains a resin (B) and a resin (C) in it as an indispensable component, after sinking in at the time of heat can also be adopted.

[0025] As a distributed assistant, although the protective colloid of well-known common use, a surfactant, etc. can be used, a polyethylene oxide, polyoxyalkylene monochrome phenol ether, an alkylbenzene sulfonic-acid metal salt, polyvinyl alcohol, self-emulsifiability acrylic resin, a self-emulsifiability epoxy resin, etc. can be used, for example.

[0026] Moreover, it is desirable to use various macromolecule fixing agents for it, in order to raise the yield at the time of paper making to resin powder. As these fixing agents, a polyacrylamide resin, an aluminum sulfate, a polyaluminium chloride, etc. are mentioned, for example.

[0027] In obtaining the dry prepreg sheet, methods, such as hot air drying, far-infrared irradiation, and microwave irradiation, are employable, for example. Drying until only the adhesiveness of the sheet itself is lost is desirable. If it dries highly, it will become difficult to carry out an allocated type to the configuration of the request to the below-mentioned compression molding. In obtaining a precise carbonaceous fabrication board, it carries out by examining this dryness condition.

[0028] Although the thing of what thickness is sufficient as the prepreg sheet of this invention, it is a 0.2-1.2mm thing, for example. The weights per two are usually 75-300g 1m.

[0029] Thus, let the obtained sheet be for example, a porous carbonaceous fabrication board by calcinating, after pressing and carrying out an allocated type. In addition in this compression molding, fabrication is simultaneously performed also for heat if needed using the metal mold of well-known common use. of course, metal mold -- vacuum compression forming which deaerates and fabricates inside to a vacuum is also employable

[0030] Also in which method, in a sheet, although two or more sheet laminating is carried out and being fabricated, it is also possible in this case one sheet or to carry out the laminating of the sheet of a different kind to it, and to fabricate it according to the purpose.

[0031] Subsequently, let the forming board obtained by the aforementioned fabrication be a carbonaceous and porous forming board by calcinating. Although especially the conditions of baking are not restricted, they can obtain a carbonaceous forming board by calcinating at the temperature of 1000-3000 degrees C, for example in inert gas atmosphere, such as nitrogen and an argon, or a vacuum. It is also possible to graphitize by calcinating at the temperature of further 2000-3000 degrees C depending on the purpose.

[0032] Thus, the obtained porous carbonaceous fabrication board is employable as the various uses of well-known common use. Specifically, the use of various electrode materials, a separator, a heat insulator, etc. is mentioned.

[0033] Using the organic fiber which is not fibrillating, the epoxy resin weight to a solid phenol resin weight is 5 - 50% of the weight of a range, and the carbonaceous fabrication board obtained using the sheet of this invention which is 5 to 50% of the weight of a range comparatively of the aforementioned organic fiber to a carbon fiber satisfies the surface smooth nature and intensity as which the property is required of an electrode board, gas air permeability, etc., and serves as a material very useful as an object for electrode boards. It is well in agreement with the specification value as a phosphoric acid type fuel cell electrode board especially.

[0034] The military requirement these-described above is peculiar to an electrode board, and it being what cannot be directly expected from the forming board which only fabricated and was obtained is that there is nothing \*\*\*\* about garrulity.

[0035]

[Example] Next, an example explains this invention in detail. The following, as long as there is no notice, each of "sections" and "%s" is taken as weight criteria.

[0036] They are ratios 4/6 (weight ratio) about the pitch based carbon fiber [DONAKABO Made from DONAKKU, and 13.5 micrometers of diameters of fiber] and polybutylene-terephthalate (PBT) fiber [1dx3mm] of the [example 1] aspect ratio 460. It distributed in water at 0.2% of sum total fiber concentration, self-hardening type phenol resin (weight average molecular weight 12000) and the bisphenol A diglycidyl ether type epoxy resin (100 degrees C of melting points, weight per epoxy equivalent 950) were added there, and the slurry was prepared.

[0037] The sheet was milled for the content of each component as the phenol resin powder 20 section, the epoxy resin powder 1 section, the carbon fiber 200 section, and the PBT fiber 10 section. The organic fiber content [ as opposed to 5 % of the weight and a carbon fiber in the epoxy resin addition to the phenol resin at this time ] was 5 % of the weight.

[0038] In order to improve the yield of resin powder, the cation nature polyacrylamide was added a total of 0.3% of opposite fiber to the above-mentioned slurry, and improvement in fixing to the fiber base material of a resinous principle was aimed at to it. Paper making of this slurry was carried out in the inclined type wire gauze nonwoven fabric manufacturing installation, and the wet web which carried out humidity was obtained.

[0039] This web was dried for 4 minutes by 140-degree C hot blast, and the prepreg sheet of 175 g/m2 of non-adhesiveness was obtained.

[0040] Two sheets of this sheet were pressed in piles (for [ 170-degree-C and pressure / of 7kg/cm ] 2 or 20 minutes), it calcinated at 2000 degrees C in the vacuum further, and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 1.

[0041] Except not using the [example 1 of comparison] epoxy resin powder 1 section, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 1.

[0042]

[Table 1]

表 1

	実施例 1	比較例 1
坪量 (g/m <sup>2</sup> )	1 7 0	1 6 8
密度 (g/cm <sup>3</sup> )	0. 2 8	0. 2 7
厚み (mm)	0. 4 2	0. 4 2
かさ密度 (g/cm <sup>3</sup> )	0. 4 2	0. 4 2
曲げ強度 (kgf/mm <sup>2</sup> )	1. 5	1. 4
黒鉛板の表面状態 (目視)	○	×
磷酸型燃料電池電極板としてのガス透気度	○	○

[0043] The actual measurement conditions of these evaluation criteria are as follows.

Basis weight: JIS It measured based on P8124.

Density: JIS It measured based on P8118.

Thickness: Measure using a micrometer.

Bulk density: JIS It measured based on R7212.

Flexural strength: Same as the above.

Surface state (surface smooth nature): It judged visually.

The degree of gas infiltration (fault): It measured with improvement Gurley method.

[0044] Since the prepreg of an example 1 is using the epoxy resin together, according to the plasticization effect of that, the dispersibility and the fluidity of a resinous principle are good at the time of thermoforming, and the homogeneous forming board is obtained at it, as contrast with an example 1 and the example 1 of comparison shows. Therefore, it turns out that the graphite board which calcinated it is also the thing excellent in surface smooth nature.

[0045] Except using it, the slurry as a publication was prepared according to having described above respectively to the [examples 2-3] table 2, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 2.

[0046] In addition, using the same thing as having used in the example 1, using the DAIWABO rayon (1dx3mm) by Daiwabo Co., Ltd. as rayon fiber, as wood pulp fiber, phenol resin, the epoxy resin, and the carbon fiber were exposed, and, on the other hand, used pulp (broad-leaved tree kraft pulp and freeness 600cc).

[0047]

[Table 2]

表 2

		単位	実施例 2	実施例 3
樹脂粉末	(B) フェノール樹脂	部	8 5	
	(C) エポキシ樹脂	部	2 0	
	(C)/(B)×100	%	2 4	
繊維基材	(A) 炭素繊維	部	6 0	
	(D) 有機繊維 種類 含有量	部	レヨン繊維 1 0	木材ハル 1 0
	(D)/(A)×100	%	1 7	
シート物性	坪 量	g/m <sup>2</sup>	1 7 5	1 7 6
	密 度	g/cm <sup>3</sup>	0. 3 4	0. 3 4
黒鉛板の物性	厚 み	mm	0. 4 0	0. 4 0
	かさ密度	g/cm <sup>3</sup>	0. 5 1	0. 5 1
	曲げ強度	kgf/mm <sup>2</sup>	2. 0	2. 0
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	×

—phenol  
—epoxy

[0048] Although the graphite board of an example 3 had and carried out gas infiltration, it was inadequate as an object for phosphoric acid type fuel cell electrode boards.

[0049] Except using it, the slurry as a publication was prepared according to having described above respectively to the [examples 4-5] table 3, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 3.

[0050] In addition, on the other hand, rayon fiber used 1dx3mm \*\* as polypropylene (PP) fiber using the same thing as having used in the example 2 using the same thing as having used phenol resin, the epoxy resin, and the carbon fiber in the example 1.

[0051]

[Table 3]

表 3

		単位	実施例 4	実施例 5
樹脂粉末	(B) フェノール樹脂	部	85	100
	(C) エポキシ樹脂	部	42	20
	(C)/(B)×100	%	49	20
繊維基材	(A) 炭素繊維	部	60	15
	(D) 有機繊維 種類 含有量	部	PP 繊維 30	レヨン繊維 3
	(D)/(A)×100	%	50	20
シート物性	坪量 密度	g/m <sup>2</sup> g/cm <sup>2</sup>	160 0.35	174 0.40
黒鉛板の物性	厚み	mm	0.39	0.40
	かさ密度	g/cm <sup>3</sup>	0.52	0.60
	曲げ強度	kgf/mm <sup>2</sup>	1.9	1.3
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	○

[0052]

[Effect of the Invention] In this invention, since the epoxy resin is used together in obtaining the prepreg sheet for porous carbonaceous fabrication boards to the phenol resin which is the conventional resinous principle, the exceptional remarkable effect that the porous carbonaceous fabrication board of the surface smooth nature which was finally excellent is obtained is done so.

[Translation done.]

## \* NOTICES \*

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## EXAMPLE

[Example] Next, an example explains this invention in detail. The following, as long as there is no notice, each of "sections" and "%s" is taken as weight criteria.

[0036] They are ratios 4/6 (weight ratio) about the pitch based carbon fiber [DONAKABO Made from DONAKKU, and 13.5 micrometers of diameters of fiber] and polybutylene-terephthalate (PBT) fiber [1dx3mm] of the [example 1] aspect ratio 460. It distributed in water at 0.2% of sum total fiber concentration, self-hardening type phenol resin (weight average molecular weight 12000) and the bisphenol A diglycidyl ether type epoxy resin (100 degrees C of melting points, weight per epoxy equivalent 950) were added there, and the slurry was prepared.

[0037] The sheet was milled for the content of each component as the phenol resin powder 20 section, the epoxy resin powder 1 section, the carbon fiber 200 section, and the PBT fiber 10 section. The organic fiber content [ as opposed to 5 % of the weight and a carbon fiber in the epoxy resin addition to the phenol resin at this time ] was 5 % of the weight.

[0038] In order to improve the yield of resin powder, the cation nature polyacrylamide was added a total of 0.3% of opposite fiber to the above-mentioned slurry, and improvement in fixing to the fiber base material of a resinous principle was aimed at to it. Paper making of this slurry was carried out in the inclined type wire gauze nonwoven fabric manufacturing installation, and the wet web which carried out humidity was obtained.

[0039] This web was dried for 4 minutes by 140-degree C hot blast, and the prepreg sheet of 175 g/m2 of non-adhesiveness was obtained.

[0040] Two sheets of this sheet were pressed in piles (for [ 170-degree-C and pressure / of 7kg/cm ] 2 or 20 minutes), it calcinated at 2000 degrees C in the vacuum further, and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 1.

[0041] Except not using the [example 1 of comparison] epoxy resin powder 1 section, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 1.

[0042]

[Table 1]

表 1

	実施例 1	比較例 1
坪量 (g/m <sup>2</sup> )	170	168
密度 (g/cm <sup>3</sup> )	0.28	0.27
厚み (mm)	0.42	0.42
かさ密度 (g/cm <sup>3</sup> )	0.42	0.42
曲げ強度 (kgf/mm <sup>2</sup> )	1.5	1.4
黒鉛板の表面状態 (目視)	○	×
磷酸型燃料電池電極板としてのガス透気度	○	○

[0043] The actual measurement conditions of these evaluation criteria are as follows.

Basis weight: JIS It measured based on P8124.

Density: JIS It measured based on P8118.

Thickness: Measure using a micrometer.



Bulk density: JIS It measured based on R7212.

Flexural strength: Same as the above.

Surface state (surface smooth nature): It judged visually.

The degree of gas infiltration (fault): It measured with improvement Gurley method.

[0044] Since the prepreg of an example 1 is using the epoxy resin together, according to the plasticization effect of that, the dispersibility and the fluidity of a resinous principle are good at the time of thermoforming, and the homogeneous forming board is obtained at it, as contrast with an example 1 and the example 1 of comparison shows. Therefore, it turns out that the graphite board which calcinated it is also the thing excellent in surface smooth nature.

[0045] Except using it, the slurry as a publication was prepared according to having described above respectively to the [examples 2-3] table 2, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 2.

[0046] In addition, using the same thing as having used in the example 1, using the DAIWABO rayon (1dx3mm) by Daiwabo Co., Ltd. as rayon fiber, as wood pulp fiber, phenol resin, the epoxy resin, and the carbon fiber were exposed, and, on the other hand, used pulp (broad-leaved tree kraft pulp and freeness 600cc).

[0047]

[Table 2]

表 2

		単位	実施例 2	実施例 3
樹脂粉末	(B) フェノール樹脂	部	8 5	
	(C) エポキシ樹脂	部	2 0	
	(C)/(B)×100	%	2 4	
繊維基材	(A) 炭素繊維	部	6 0	
	(D) 有機繊維 種類 含有量	部	レーヨン繊維 1 0	木材パルプ 1 0
	(D)/(A)×100	%	1 7	
シート物性	坪量	g/m <sup>2</sup>	1 7 5	1 7 6
	密度	g/cm <sup>3</sup>	0. 3 4	0. 3 4
黒鉛板の物性	厚み	mm	0. 4 0	0. 4 0
	かさ密度	g/cm <sup>3</sup>	0. 5 1	0. 5 1
	曲げ強度	kgf/mm <sup>2</sup>	2. 0	2. 0
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	×

[0048] Although the graphite board of an example 3 had and carried out gas infiltration, it was inadequate as an object for phosphoric acid type fuel cell electrode boards.

[0049] Except using it, the slurry as a publication was prepared according to having described above respectively to the [examples 4-5] table 3, paper making was performed on the same conditions as an example 1, the prepreg sheet was obtained, it calcinated on the conditions same subsequently and the graphite board was obtained. The physical-properties value of the obtained graphite board was shown in Table 3.

[0050] In addition, on the other hand, rayon fiber used 1dx3mm \*\* as polypropylene (PP) fiber using the same thing as having used in the example 2 using the same thing as having used phenol resin, the epoxy resin, and the carbon fiber in the example 1.

[0051]

[Table 3]

表 3

		単位	実施例 4	実施例 5
樹脂粉末	(B) フェノール樹脂	部	85	100
	(C) エポキシ樹脂	部	42	20
	(C)/(B)×100	%	49	20
繊維基材	(A) 炭素繊維	部	60	15
	(D) 有機繊維 種類 含有量	部	PP 繊維 30	レヨン繊維 3
	(D)/(A)×100	%	50	20
シート物性	坪量	g/m <sup>2</sup>	160	174
	密度	g/cm <sup>3</sup>	0.35	0.40
黒鉛板の物性	厚み	mm	0.39	0.40
	かさ密度	g/cm <sup>3</sup>	0.52	0.60
	曲げ強度	kgf/mm <sup>2</sup>	1.9	1.3
	表面状態 (目視)		○	○
	磷酸型燃料電池電極板としてのガス透気度		○	○

[Translation done.]